

Synopsis

NiTi based shape memory alloys (SMA) cover most of the commercially produced shape memory devices and components. The reversible martensitic transformation between the phases B2 (austenite) and B19' (martensite) is responsible for the shape memory effect in these alloys. The amount of strain which can be regained after a permanent deformation through thermal activation, known as the recoverable strain, is a strong function of crystallographic texture and microstructure. Texture influences the activation of a specific martensite variant during stress induced martensitic (SIM) transformation and also the re-orientation of twinned variants during further deformation. The variant selection decides the amount of recoverable strain. Since the NiTi based shape memory alloys inevitably undergo thermo-mechanical processing in the course of component design, the consequent evolution of texture and microstructure regulate the shape memory behavior. The present thesis is aimed to address this issue in some NiTi alloys that are technologically important for different applications, namely a binary Ni-rich NiTi alloy, a copper containing NiTi alloy and a hafnium containing NiTi alloy. The Ni rich NiTi alloy displays pseudoelastic behavior that can be used for couplings, the NiTiCu alloy provides a controlled thermal hysteresis suitable for actuator applications and the NiTiHf alloy can be used for high temperature applications.

The first Chapter of the thesis provides a detailed overview of the existing knowledge related to evolution of microstructure and texture during processing, the transformation texture and its role on the shape memory behavior in NiTi alloys. The second chapter includes the experimental procedure followed to generate different textures, namely unidirectional and cross rolling with and without a subsequent annealing and also the details of the techniques used to characterize the structure, microstructure, texture and mechanical properties.

The evolution of texture during thermo-mechanical processing of a Ni rich NiTi alloy and its impact on shape memory behavior is addressed in Chapter 3. The two modes of rolling employed at higher temperature led to the formation of different textures. The texture of unidirectionally rolled samples was characterized by a strong $\langle 111 \rangle$ ||ND fiber, while a strong Goss $\{100\}$ $\langle 110 \rangle$ component along with $\langle 111 \rangle$ ||ND fiber was observed in

the texture of the cross rolled samples. Annealing of the unidirectionally rolled samples generated a strong $\langle 100 \rangle \parallel \text{ND}$ fiber, and a weak $\langle 111 \rangle \parallel \text{ND}$ fiber was observed for the cross rolled samples. Microtexture analyses indicated that dynamically recrystallized grains had significantly different texture compared to the statically annealed material. One of the salient features of this study is the analysis of different twin boundaries with coincident site lattice (CSL) relations that has been observed in the hot rolled material. The origin of these twins has been attributed to deformation. The evolution of twin boundaries with CSL relation has strong influence on texture formation. A few of the important texture components have been found to have CSL relation amongst them. The origin of different texture components were found using intra-grain misorientation parameters.

In-situ transformation studies in a scanning electron microscope have confirmed the formation of different types of twins at very low amount of strain in the Ni rich NiTi alloy. A Schmid factor based criterion was used to identify the activation of a particular variant. Trace analysis of the surface relief due to SIM transformation was utilized to confirm the theoretically predicted variant. Schmid criterion has been found to be valid in all the cases. Modulus variation with temperature and strain was studied using dynamical mechanical analysis. Microstructural changes during thermal and thermo-mechanical cycling revealed higher orientation gradient along grain boundaries compared to grain interior. The compatibility condition at the grain boundaries were attributed to higher misorientation development. Misorientation development during cycling loading process is also found to be a strong function of texture. Processing condition and texture has a strong influence on the recoverable strain. Particularly, the strength of $\langle 111 \rangle \parallel \text{ND}$ fiber is influential in deciding the recoverable strain.

Study of microstructure and texture evolution in the TiNiCu SMA and subsequent study on its impact on recoverable strain is presented in Chapter 4. Convincing evidences for the mechanisms operating during different dynamic restoration processes have been presented through microstructural investigation. Texture analysis of the austenite phase showed the formation of $\langle 111 \rangle \parallel \text{ND}$ fiber. Despite the weakening of texture at larger strain, strength of certain deformation texture components like S $\{123\} \langle 634 \rangle$ and Cu $\{112\} \langle 111 \rangle$ increased, which suggested that texture evolution in TiNiCu alloy deviates

from the texture of binary NiTi at large strains. Transformation texture analysis was carried out through electron back scattered diffraction technique, using an in-situ heating stage. The analysis of the results showed predominant activation of $\langle 011 \rangle$ type II as well as $\{11\bar{1}\}$ type I twins. A comparison of martensite and austenite pole figures indicated strong variant selection during phase transformation. Like the binary NiTi alloy, cross rolling of TiNiCu alloy also showed ample changes in the texture of martensite phase through the formation of different texture components. Annealing of both unidirectionally and cross rolled samples led to the weakening of texture. The change in volume fraction of Ti_2NiCu precipitates, resulting from different processing conditions, influenced the transformation temperature. In this case also, texture and large intra-grain misorientation governed the recoverable strain.

Chapter 5 is dedicated to the study of high temperature NiTiHf alloy. X-ray diffraction and differential scanning calorimetric studies confirmed a two step martensitic transformation, a B19' monoclinic and rhombohedral R-phase martensite in the studied alloy ($\text{Ni}_{49.4}\text{Ti}_{38.6}\text{Hf}_{12}$). Microstructural investigations showed the formation of dendritic $(\text{Ti,Hf})_2\text{Ni}$ precipitates along the grain boundary. Evolution of R-phase martensite was always observed along with $(\text{Ti,Hf})_2\text{Ni}$ precipitates, irrespective of the processing condition. Dissolution of $(\text{Ti,Hf})_2\text{Ni}$ precipitates by solution treatment suppressed the R phase formation. Strong texture of R-phase martensite confirmed variant selection during martensitic transformation. On the contrary, texture of B19' martensite was always weak, suggesting no preference for variant selection. Rolled material with a relatively strong texture exhibited higher recoverable strain compared to annealed material.

Finally, all the significant outcomes of the present investigation are summarized in Chapter 6. Based on the conclusions, suggestions for future work have been mentioned.